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FILE 'HOME' ENTERED AT 09:56:42 ON 25 OCT 2007

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L11 ANSWER 1 OF 4 CA COPYRIGHT 2007 ACS on STN
 ACCESSION NUMBER: 146:295733 CA <<LOGINID::20071025>>
 TITLE: N-Oxidation of pyridine carboxylic acids using
 hydrogen peroxide catalyzed by a green
 heteropoly acid catalyst: Preyssler's anion,
 [NaP5W30O110]14-
 AUTHOR(S): Bamoharram, Fatemeh F.; Heravi, Majid M.; Roshani,
 Mina; Tavakoli, Niloofar
 CORPORATE SOURCE: Department of Chemistry, Islamic Azad
 University-Mashhad Branch, Mashhad, Iran
 SOURCE: Journal of Molecular Catalysis A: Chemical (2006),
 252(1-2), 219-225
 CODEN: JMCCF2; ISSN: 1381-1169
 PUBLISHER: Elsevier B.V.
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 OTHER SOURCE(S): CASREACT 146:295733
 AB The catalytic oxidn. of pyridine carboxylic acids such as nicotinic acid, picolinic acid and quinolinic acid to the corresponding N-oxides has been studied using hydrogen peroxide as oxidant and Preyssler's catalyst as H14 [NaP5W30O110] and H14 [NaP5W29MoO110]. The highly selective oxidns. gave good yields of the related N-oxides along with decarboxylation of the amines. In the prodn. of pyridine N-oxides, the position of COOH group is found to play an important role in detg. the product type, and decarboxylation takes place only at the 2-position to nitrogen. The oxidn. reactions were extended to other tertiary amines.

REFERENCE COUNT: 64 THERE ARE 64 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE REFORMAT.

L11 ANSWER 2 OF 4 CA COPYRIGHT 2007 ACS on STI

ACCESSION NUMBER: 143:117138 CA <<LOGINID::20071025>>

TITLE: Catalyst for synthesis of 2- and 4-picoline, process for preparing 2- and 4-picoline and process for preparing the catalyst

INVENTOR(S): Dutta, Pashupati; Roy, Subhash Chandra; Roy, Shyam Kishor; Goswami, Tarun Kanti

PATENT ASSIGNEE(S): Council of Scientific & Industrial Research, India
SOURCE: PCT Int. Appl., 10 pp.

CODEN: PIXXDJ

DOCUMENT TYPE: Patent

LANGUAGE : English

FAMILY ACC. NUM. CO

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2005063389	A1	20050714	WO 2003-IN467	20031231
W:	AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW			
RW:	BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK			

TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG
 CA 2552158 A1 20050714 CA 2003-2552158 20031231
 AU 2003300723 A1 20050721 AU 2003-300723 20031231
 EP 1708811 A1 20061011 EP 2003-819218 20031231
 R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,
 IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, SK
 CN 1886195 A 20061227 CN 2003-80110944 20031231
 IN 2004DN00489 A 20060310 IN 2004-DN489 20040227
 US 2005209458 A1 20050922 US 2004-806063 20040322
 WO 2003-IN467 W 20031231

PRIORITY APPLN. INFO.:

CASREACT 143:117138

OTHER SOURCE(S):
AB Title catalyst comprises a ***heteropoly*** acid selected from the group consisting of ***silicotungstic*** acid, ***phosphotungstic*** acid, ***phosphomolybdic*** acid and ***vanadotungstic*** acid provided on a ***support***. The ***support*** is selected from the group consisting of silica gel, alumina, silica-alumina, clays and montmorillonite. The invention also provides a process for the prepn. thereof and use thereof for the synthesis of 2- and 4-picoline useful as intermediates for pharmaceuticals and agrochems.

REFERENCE COUNT: 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L11 ANSWER 3 OF 4 CA COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 106:86707 CA <<LOGINID::20071025>>

TITLE: Picoline manufacture by catalytic vapor-phase cyclocondensation of ethanol with ammonia

INVENTOR(S): Rajaram, Potaraju; Joshi, Milind Vishnu

PATENT ASSIGNEE(S): IEL Ltd., India

SOURCE: Eur. Pat. Appl., 9 pp.
CODEN: EPXXDW

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 209241	A2	19870121	EP 1986-304428	19860610
EP 209241	A3	19880120		

R: GB

IN 164212 A1 19890204 IN 1985-CA218 19850325

GB 1985-14611 A 19850610

PRIORITY APPLN. INFO.:
AB Pyridines are prepd. from EtOH and NH₃ in the vapor phase in the presence of ***heteropolyacid*** /.gamma.-alumina catalysts at 350-500.degree.. Spraying 100 g .gamma.-alumina granules with an 8.6mM MeCN soln. of H₃PMo₁₂O₄₀ (I), drying in air for 12 h and at 110.degree. for 2 h and activating in a stream of air at 500.degree. for 2 h gave a acatalyst contg. 20% I. The prepd. catalyst contained 20% 12-molybdochosphoric acid, and was packed into a tubular reactor, the temp. of which was gradually raised to 400.degree.. Passing 0.05 mL/min EtOH and 40 mL/min NH₃ over this catalyst at 400.degree. gave a 40% conversion of EtOH to 2- and 4-picoline with >55 mol% selectivity.

L11 ANSWER 4 OF 4 CA COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 89:43139 CA <<LOGINID::20071025>>

TITLE: Pyridine bases

INVENTOR(S): Miyake, Tetsuya; Noguchi, Kohji; Imamura, Kazuo

PATENT ASSIGNEE(S): Asahi Chemical Industry Co., Ltd., Japan

SOURCE: Ger. Offen., 50 pp.
CODEN: GWXXBX

DOCUMENT TYPE: Patent

LANGUAGE: German

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 2746177	A1	19780503	DE 1977-2746177	19771014
DE 2746177	C2	19831229		
JP 53053658	A	19780516	JP 1976-128192	19761027
JP 53053660	A	19780516	JP 1976-128194	19761027
JP 60016418	B	19850425		

This
and

JP 53053661	A	19780516	JP 1976-128195	19761027
JP 60032627	B	19850729		
US 4179576	A	19791218	US 1977-841083	19771011
GB 1579473	A	19801119	GB 1977-42496	19771012
FR 2369265	A1	19780526	FR 1977-31023	19771014
FR 2369265	B1	19800627		
CH 633777	A5	19821231	CH 1977-12608	19771014
CA 1094562	A1	19810127	CA 1977-288953	19771018
US 4263439	A	19810421	US 1979-48572	19790614
US 4239892	A	19801216	US 1979-61335	19790727
PRIORITY APPLN. INFO.:			JP 1976-128192	A 19761027
			JP 1976-128194	A 19761027
			JP 1976-128195	A 19761027
			US 1977-841083	A3 19771011

OTHER SOURCE(S): MARPAT 89:43139

AB Pyridine bases were prep'd. by heating AcH with NH₃ over transition metal oxides and ***heteropoly*** oxides on Al₂O₃ or SiO₂ ***supports***. Thus, a 1-1.5 AcH-NH₃ mixt. at 430.degree. and 1000 h⁻¹ space velocity over a catalyst prep'd. by gelling Al₂(SO₄)₃ with ammonium paratungstate followed calcining gave 69.0% pyridine bases including 2- and 4-methylpyridine.

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L13 ANSWER 1 OF 3 CA COPYRIGHT 2007 ACS on STN
 ACCESSION NUMBER: 134:312754 CA <<LOGINID::20071025>>
 TITLE: Synthesis of 3-picoline-1-oxide catalyzed by ***phosphotungstic*** acid ***supported*** on activated carbon
 AUTHOR(S): Zhang, Heng
 CORPORATE SOURCE: Jinxi Research Institute of Chemical Industry, Huludao, 125001, Peop. Rep. China
 SOURCE: Jingxi Huagong (2000), 17(11), 676-678
 PUBLISHER: CODEN: JIHUFJ; ISSN: 1003-5214
 DOCUMENT TYPE: Journal
 LANGUAGE: Chinese
 AB 3-Picoline-1-oxide (I) was prep'd. by oxidn. of 3-picoline with H₂O₂ and the effects of various factors on the reaction were discussed. The optimal conditions, appropriate for scale-up were: the mol. ratio H₂O₂/3-picoline 1.2:1.0, reaction time 5 h, the wt. percentage of catalyst ***supported*** on active C 18%. The yield of I reached 92.0%.

L13 ANSWER 2 OF 3 CA COPYRIGHT 2007 ACS on STN
 ACCESSION NUMBER: 111:77817 CA <<LOGINID::20071025>>
 TITLE: Catalytic oxidation systems. II. Formation of N-oxides of pyridine, alkylpyridines, and pyridinecarboxylic acids
 AUTHOR(S): Cabre Castellvi, Juan; Palomo Coll, Alberto
 CORPORATE SOURCE: Dep. Invest., Gema S. A., Barcelona, 08021, Spain
 SOURCE: Afinidad (1988), 45(418), 511-15
 DOCUMENT TYPE: CODEN: AFINAE; ISSN: 0001-9704
 LANGUAGE: Spanish
 AB N-Oxidn. of the title pyridines was examd. using systems comprising H₂O₂ as oxidant and ***phosphotungstic***, ***phosphomolybdic***, and ***silicotungstic*** acids as catalysis. ***Phosphotungstic*** acid is an efficient catalyst at room temp.

L13 ANSWER 3 OF 3 CA COPYRIGHT 2007 ACS on STN
 ACCESSION NUMBER: 100:51464 CA <<LOGINID::20071025>>
 TITLE: N-Oxides of pyridine and quinoline derivatives
 INVENTOR(S): Nagy, Ferenc; Lošonczi, Bela; Lengyel, Meszaros Agnes; Kovacs, Istvan; Havasi, Gabor; Felmeri, Jozsef; Rusznak, Istvan; Kormondi, Istvan
 PATENT ASSIGNEE(S): Richter Gedeon Vegyeszeti Gyar Rt., Hung.
 SOURCE: Hung. Teljes, 14 pp.
 DOCUMENT TYPE: Patent
 LANGUAGE: Hungarian

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
HU 25878	A2	19830829	HU 1981-2813	19810930
HU 183595	B	19840528		
PRIORITY APPLN. INFO.:			HU 1981-2813	19810930
GI				

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AB Pyridine and quinoline N-oxides (I and II, resp.) (R1 and R2 = H, Cl-4 alkyl, halo, CO₂H, CN, or CONH₂) are prep'd. from the corresponding parent compds. by oxidn. with H₂O₂ in the presence of ***phosphomolybdic*** or ***phosphotungstic*** acid catalysts. Thus, 10 g pyridine and 0.5 g ***phosphotungstic*** acid in 5 mL water was treated with 17.1 g 30% H₂O₂, at 80.degree., followed by heating to 90.degree. for 1 h to give 15 g pyridine N-oxide-HCl.

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(FILE 'HOME' ENTERED AT 09:56:42 ON 25 OCT 2007)

FILE 'REGISTRY' ENTERED AT 09:56:57 ON 25 OCT 2007

L1 0 S SILOCOTUNG?

FILE 'CA' ENTERED AT 09:57:14 ON 25 OCT 2007

L2 23175 S PICOLIN?

L3 0 S L2/PREP

FILE 'REGISTRY' ENTERED AT 09:57:35 ON 25 OCT 2007

L4 6110 S PICOLINE

FILE 'CA' ENTERED AT 09:57:54 ON 25 OCT 2007

L5 34319 S L4

L6 6636 S L4/PREP

L7 34175 S SILICOTUN? OR PHOSPHOT? OR PHOSPHOMOL? OR VANADOTUN?

L8 762195 S SUPPORT?

L9 794542 S L7 OR L8

L10 181 S L9 AND L6

L11 4 S L10 AND HETEROPOLY?

FILE 'STNGUIDE' ENTERED AT 10:00:11 ON 25 OCT 2007

FILE 'CA' ENTERED AT 10:02:16 ON 25 OCT 2007

L12 6 S L7 AND L10

L13 3 S L12 NOT L11

FILE 'STNGUIDE' ENTERED AT 10:03:27 ON 25 OCT 2007

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SESSION WILL BE HELD FOR 120 MINUTES

STN INTERNATIONAL SESSION SUSPENDED AT 10:04:37 ON 25 OCT 2007

10/806063

ACCESSION NUMBER: 143:117138 CA
TITLE: Catalyst for synthesis of 2- and 4-picoline, process
for preparing 2- and 4-picoline and process for
preparing the catalyst
INVENTOR(S): Dutta, Pashupati; Roy, Subhash Chandra; Roy, Shyam
Kishor; Goswami, Tarun Kanti
PATENT ASSIGNEE(S): Council of Scientific & Industrial Research, India
SOURCE: PCT Int. Appl., 10 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2005063389	A1	20050714	WO 2003-IN467	20031231
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW				
RW: BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
CA 2552158	A1	20050714	CA 2003-2552158	20031231
AU 2003300723	A1	20050721	AU 2003-300723	20031231
EP 1708811	A1	20061011	EP 2003-819218	20031231
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, SK				
CN 1886195	A	20061227	CN 2003-80110944	20031231
IN 2004DN00489	A	20060310	IN 2004-DN489	20040227
US 2005209458	A1	20050922	US 2004-806063	20040322
PRIORITY APPLN. INFO.:			WO 2003-IN467	W 20031231

OTHER SOURCE(S): CASREACT 143:117138

AB Title catalyst comprises a heteropoly acid selected from the group consisting of silicotungstic acid, phosphotungstic acid, phosphomolybdic acid and vanadotungstic acid provided on a support. The support is selected from the group consisting of silica gel, alumina, silica-alumina, clays and montmorillonite. The invention also provides a process for the preparation thereof and use thereof for the synthesis of 2- and 4-picoline useful as intermediates for pharmaceuticals and agrochems.

REFERENCE COUNT: 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

10/806063

ACCESSION NUMBER: 106:86707 CA
TITLE: Picoline manufacture by catalytic vapor-phase
cyclocondensation of ethanol with ammonia
INVENTOR(S): Rajaram, Potaraju; Joshi, Milind Vishnu
PATENT ASSIGNEE(S): IEL Ltd., India
SOURCE: Eur. Pat. Appl., 9 pp.
CODEN: EPXXDW
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 209241	A2	19870121	EP 1986-304428	19860610
EP 209241	A3	19880120		
R: GB				
IN 164212	A1	19890204	IN 1985-CA218	19850325
PRIORITY APPLN. INFO.:			GB 1985-14611	A 19850610

AB Pyridines are prepared from EtOH and NH₃ in the vapor phase in the presence of heteropolyacid/γ-alumina catalysts at 350-500°. Spraying 100 g γ-alumina granules with an 8.6mM MeCN solution of H₃PMo₁₂O₄₀ (I), drying in air for 12 h and at 110° for 2 h and activating in a stream of air at 500° for 2 h gave a catalyst containing 20% I. The prepared catalyst contained 20% 12-molybdophosphoric acid, and was packed into a tubular reactor, the temperature of which was gradually raised to 400°. Passing 0.05 mL/min EtOH and 40 mL/min NH₃ over this catalyst at 400° gave a 40% conversion of EtOH to 2- and 4-picoline with >55 mol% selectivity.

10/806063

ACCESSION NUMBER: 89:43139 CA
TITLE: Pyridine bases
INVENTOR(S): Miyake, Tetsuya; Noguchi, Kohji; Imamura, Kazuo
PATENT ASSIGNEE(S): Asahi Chemical Industry Co., Ltd., Japan
SOURCE: Ger. Offen., 50 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 2746177	A1	19780503	DE 1977-2746177	19771014
DE 2746177	C2	19831229		
JP 53053658	A	19780516	JP 1976-128192	19761027
JP 53053660	A	19780516	JP 1976-128194	19761027
JP 60016418	B	19850425		
JP 53053661	A	19780516	JP 1976-128195	19761027
JP 60032627	B	19850729		
US 4179576	A	19791218	US 1977-841083	19771011
GB 1579473	A	19801119	GB 1977-42496	19771012
FR 2369265	A1	19780526	FR 1977-31023	19771014
FR 2369265	B1	19800627		
CH 633777	A5	19821231	CH 1977-12608	19771014
CA 1094562	A1	19810127	CA 1977-288953	19771018
US 4263439	A	19810421	US 1979-48572	19790614
US 4239892	A	19801216	US 1979-61335	19790727
PRIORITY APPLN. INFO.:			JP 1976-128192	A 19761027
			JP 1976-128194	A 19761027
			JP 1976-128195	A 19761027
			US 1977-841083	A3 19771011

OTHER SOURCE(S): MARPAT 89:43139
AB Pyridine bases were prepared by heating AcH with NH₃ over transition metal oxides and heteropoly oxides on Al₂O₃ or SiO₂ supports. Thus, a 1-1.5 AcH-NH₃ mixture at 430° and 1000 h⁻¹ space velocity over a catalyst prepared by gelling Al₂(SO₄)₃ with ammonium paratungstate followed calcining gave 69.0% pyridine bases including 2- and 4-methylpyridine.

10/806063

ACCESSION NUMBER:

134:312754 CA

TITLE:

Synthesis of 3-picoline-1-oxide catalyzed by phosphotungstic acid supported on activated carbon

AUTHOR(S):

Zhang, Heng

CORPORATE SOURCE:

Jinxi Research Institute of Chemical Industry,
Huludao, 125001, Peop. Rep. China

SOURCE:

Jingxi Huagong (2000), 17(11), 676-678

CODEN: JIHUFJ; ISSN: 1003-5214

PUBLISHER:

Jingxi Huagong Bianjibu

DOCUMENT TYPE:

Journal

LANGUAGE:

Chinese

AB 3-Picoline-1-oxide (I) was prepared by oxidation of 3-picoline with H₂O₂ and the effects of various factors on the reaction were discussed. The optimal conditions, appropriate for scale-up were: the mol. ratio H₂O₂/3-picoline 1.2:1.0, reaction time 5 h, the weight percentage of catalyst supported on active C 18%. The yield of I reached 92.0%.

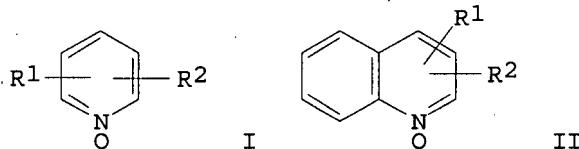
10/806063

ACCESSION NUMBER: 111:77817 CA
TITLE: Catalytic oxidation systems. II. Formation of N-oxides
of pyridine, alkylpyridines, and pyridinecarboxylic
acids
AUTHOR(S): Cabre Castellvi, Juan; Palomo Coll, Alberto
CORPORATE SOURCE: Dep. Invest., Gema S. A., Barcelona, 08021, Spain
SOURCE: Afinidad (1988), 45(418), 511-15
CODEN: AFINAE; ISSN: 0001-9704
DOCUMENT TYPE: Journal
LANGUAGE: Spanish
AB N-Oxidation of the title pyridines was examined using systems comprising H₂O₂
as oxidant and phosphotungstic, phosphomolybdic, and
silicotungstic acids as catalysis. Phosphotungstic acid
is an efficient catalyst at room temperature

10/806063

ACCESSION NUMBER: 100:51464 CA
TITLE: N-Oxides of pyridine and quinoline derivatives
INVENTOR(S): Nagy, Ferenc; Losonczi, Bela; Lengyel, Meszaros Agnes;
Kovacs, Istvan; Havasi, Gabor; Felmeri, Jozsef;
Rusznak, Istvan; Kormondi, Istvan
PATENT ASSIGNEE(S): Richter Gedeon Vegyeszeti Gyar Rt., Hung.
SOURCE: Hung. Teljes, 14 pp.
CODEN: HUXXBU
DOCUMENT TYPE: Patent
LANGUAGE: Hungarian
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
HU 25878	A2	19830829	HU 1981-2813	19810930
HU 183595	B	19840528		
PRIORITY APPLN. INFO.:			HU 1981-2813	19810930
GI				



AB Pyridine and quinoline N-oxides (I and II, resp.) (R¹ and R² = H, Cl-4 alkyl, halo, CO₂H, CN, or CONH₂) are prepared from the corresponding parent compds. by oxidation with H₂O₂ in the presence of phosphomolybdic or phosphotungstic acid catalysts. Thus, 10 g pyridine and 0.5 g phosphotungstic acid in 5 mL water was treated with 17.1 g 30% H₂O₂, at 80°, followed by heating to 90° for 1 h to give 15 g pyridine N-oxide-HCl.